

Dilead(II) hydrogenphosphite dinitrate

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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{O}-\text{N}) = 0.009$ Å; R factor = 0.012; wR factor = 0.030; data-to-parameter ratio = 11.9.

In the title compound, $\text{Pb}_2(\text{HPO}_3)(\text{NO}_3)_2$, the two distinct Pb^{2+} ions (both with site symmetry m) adopt irregular PbO_{10} coordination polyhedra. The structure is completed by two distinct nitrate groups (in which one O atom and the N atom have m site symmetry for both ions) and an HPO_3^{2-} anion (in which one O atom and the P and H atoms have m site symmetry). The connectivity of the PbO_{10} , NO_3 and HPO_3 units in the crystal structure results in a three-dimensional network.

Related literature

For related structures, see: Ouarsal *et al.* (2005*a,b*); Vasić *et al.* (1981). For bond-valence sum calculations, see: Brown & Altermatt (1985).

Experimental

Crystal data

$\text{Pb}_2(\text{HPO}_3)(\text{NO}_3)_2$	$V = 404.94(4) \text{ \AA}^3$
$M_r = 618.4$	$Z = 2$
Orthorhombic, $Pmn2_1$	Mo $K\alpha$ radiation
$a = 5.4069(2) \text{ \AA}$	$\mu = 41.76 \text{ mm}^{-1}$
$b = 10.4079(6) \text{ \AA}$	$T = 120 \text{ K}$
$c = 7.1958(4) \text{ \AA}$	$0.25 \times 0.10 \times 0.05 \text{ mm}$

Data collection

Oxford Diffraction CCD diffractometer	6814 measured reflections
Absorption correction: analytical (<i>CrysAlis RED</i> ; Oxford Diffraction, 2008)	932 independent reflections
$T_{\min} = 0.013$, $T_{\max} = 0.156$	919 reflections with $I > 3\sigma(I)$
	$R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.012$	Only H-atom coordinates refined
$wR(F^2) = 0.030$	$\Delta\rho_{\text{max}} = 0.94 \text{ e \AA}^{-3}$
$S = 1.25$	$\Delta\rho_{\text{min}} = -0.53 \text{ e \AA}^{-3}$
932 reflections	Absolute structure: Flack (1983),
78 parameters	with 431 Friedel pairs
1 restraint	Flack parameter: 0.01 (1)

Table 1

Selected bond lengths (Å).

Pb1—O1	2.346 (6)	Pb2—O2 ^v	2.7756 (11)
Pb1—O1 ⁱ	3.042 (3)	Pb2—O3 ⁱⁱ	2.513 (4)
Pb1—O3 ⁱⁱ	2.418 (4)	Pb2—O4 ⁱⁱ	2.707 (4)
Pb1—O4 ⁱⁱⁱ	2.884 (4)	Pb2—O5 ^{vi}	2.782 (4)
Pb1—O6 ^{iv}	2.881 (7)	Pb2—O5 ^{vii}	2.948 (4)
Pb1—O6 ⁱ	3.168 (4)		

Symmetry codes: (i) $-x + \frac{3}{2}, -y + 1, z - \frac{1}{2}$; (ii) $-x + 1, y, z$; (iii) $x - \frac{1}{2}, -y + 1, z - \frac{1}{2}$; (iv) $x, y, z - 1$; (v) $x + 1, y + 1, z$; (vi) $x + \frac{1}{2}, -y + 1, z - \frac{1}{2}$; (vii) $-x, y + 1, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *JANA2006* (Petříček *et al.*, 2006); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *JANA2006*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2952).

References

- Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Brown, I. D. & Altermatt, D. (1985). *Acta Cryst.* **B41**, 244–247.
- Burla, M. C., Camalli, M., Carrozzini, B., Cascarano, G., Giacovazzo, C., Polidori, G. & Spagna, R. (2003). *J. Appl. Cryst.* **36**, 1103.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Ouarsal, R., El Bali, B., Lachkar, M., Dusek, M. & Fejfarova, K. (2005*a*). *Acta Cryst.* **E61**, i168–i170.
- Ouarsal, R., El Bali, B., Lachkar, M., Dusek, M. & Fejfarova, K. (2005*b*). *Acta Cryst.* **E61**, i171–i173.
- Oxford Diffraction (2005). *CrysAlis CCD*. Oxford Diffraction Ltd, Abingdon, England.
- Oxford Diffraction (2005). *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
- Petříček, V., Dušek, M. & Palatinus, L. (2006). *JANA2006*. Institute of Physics, Prague, Czech Republic. <http://www.xray.fzu.cz/jana/jana.html>
- Vasić, P., Prelesnik, B., Herak, R. & Čurić, M. (1981). *Acta Cryst.* **B37**, 660–662.

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Dilead(II) hydrogenphosphite dinitrate

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Comment

As part of our ongoing structural studies of metal hydrogenphosphites (Ouarsal *et al.* 2005*a,b*) we now report on the preparation and crystal structure of the title compound, (I).

The structure of (I) consists of two symmetry-independent nitrate groups and one HPO₃ hydrogenphosphite tetrahedron. The Pb atoms are located between them in two different crystallographic positions, both of them being irregularly coordinated by ten O atoms (Fig. 1). In the coordination of Pb1, shorter bonds occur causing larger bond valence sum (Brown & Altermatt, 1985) [2.028 (10) and 1.917 (6) for Pb(1)O₁₀ and Pb(2)O₁₀, respectively]. On the other hand, the mean Pb—O distance is rather larger in Pb(1)O₁₀ than in Pb(2)O₁₀, respectively 2.835 (5) Å and 2.745 (6) Å. These values are comparable to those reported in Pb(H₂PO₄)₂, 2.617 Å (Vasić *et al.*, 1981). One can distinguish the two decahedrons by their respective coordination forms: one monodentate NO₃ and one monodentate HPO₃ occurs in the coordination of Pb1, while two bidentate NO₃ contribute to that of Pb2. Accordingly, the polyhedron Pb(1)O₁₀ turns to be more distorted as Pb(2)O₁₀.

Pb₂(HPO₃)(NO₃)₂ is characterized by a three-dimensional network. Although this is not a layer structure it is convenient from the explanatory point of view to distinguish two layers as indicated in Fig. 2. The slightly rotated view of a layer along [100] (Fig. 3) reveals strips extended in the *c* directions that are not interconnected within the same layer. Their connection in the *b* direction is mediated *via* the neighbouring layer which is shifted so that its Pb atoms fall into the holes. The shortest Pb—O bonds participate in the connection of the two PbO₁₀ units and in the bond towards the HPO₃ tetrahedron. As expected, H1 atom bonded to P is not involved in any hydrogen bonding. Average P—O distance and O—P—O and H—P—O angles are 1.531 (5) Å, 110.4 (2)° and 108.6 (2)° respectively. *d*_{P—H} = 1.20 (5) Å. These values lie in the same range as the ones previously reported in known phosphites (Ouarsal *et al.* 2005*b*).

Experimental

1.6161 g lead nitrate Pb(NO₃)₂·6H₂O was dissolved in 6 ml of distilled water and added to a solution of 0.4 g phosphorous acid H₃PO₃, dissolved in 5 ml water. The mixture was stirred for 1 h at 333 K, after which time the precipitate obtained was filtered out of the solution. The filtrate was allowed to stand at room temperature until many large, colourless needles of (I) arose. The crystals were recovered by filtration and washed with a water–ethanol (80:20 v/v) mixture.

Refinement

The H atom was located in a difference map and its position and *U*_{iso} values were freely refined.

Figures

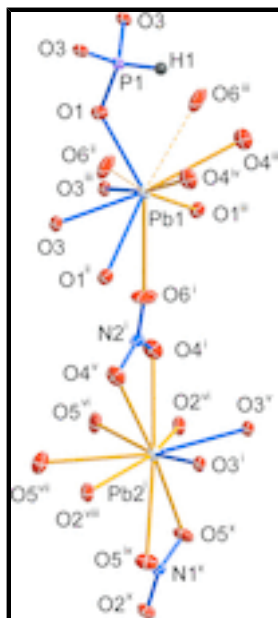


Fig. 1. A view of the asymmetric unit and some symmetry-related atoms of (I) showing 60% probability displacement ellipsoids. The various bonds lengths are distinguished by colours and style: indigo-thick up to 2.514 Å, yellow-thick from 2.707 Å to 3.042 Å, yellow-thin-dashed for 3.168 Å. [Symmetry codes: (i) $x, y, -1 + z$; (ii) $1/2 - x, 1 - y, -1/2 + z$; (iii) $3/2 - x, 1 - y, -1/2 + z$; (iv) $-1/2 - x, 1 - y, -1/2 + z$; (v) $1 - x, y, -1 + z$; (vi) $1 + x, 1 + y, -1 + z$; (vii) $-x, 1 + y, -1 + z$; (viii) $x, 1 + y, -1 + z$; (ix) $1/2 + x, 1 - y, -3/2 + z$; (x) $1/2 - x, 1 - y, -3/2 + z$]

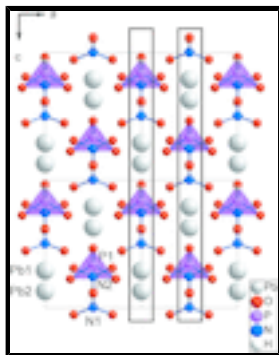


Fig. 2. A view of the structure of the title compound along [010]. The HPO_3 groups are indicated as pink tetraedra. The bonds to Pb are omitted for clarity. The black thick rectangles denote the layers that will be plotted in more details in Fig. 3.

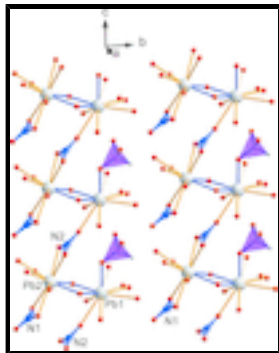


Fig. 3. The layers indicated in Fig. 2 in a view along the a direction. The first layer (grey, blue and red atoms) is plotted together with all coordinated O atoms. The various bonds lengths are distinguished by the same way as in Fig. 1.

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Crystal data

$\text{Pb}_2(\text{HPO}_3)(\text{NO}_3)_2$

$M_r = 618.4$

$F(000) = 532$

$D_x = 5.070 \text{ Mg m}^{-3}$

Orthorhombic, $Pmn2_1$
 Hall symbol: P 2ac -2
 $a = 5.4069$ (2) Å
 $b = 10.4079$ (6) Å
 $c = 7.1958$ (4) Å
 $V = 404.94$ (4) Å³
 $Z = 2$

Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
 Cell parameters from 6814 reflections
 $\theta = 3.4$ – 26.5°
 $\mu = 41.76$ mm⁻¹
 $T = 120$ K
 Plate, colourless
 $0.25 \times 0.10 \times 0.05$ mm

Data collection

Oxford Diffraction CCD
 diffractometer
 Radiation source: X-ray tube
 graphite
 Detector resolution: 8.3438 pixels mm⁻¹
 Rotation method data acquisition using ω scans
 Absorption correction: analytical
 (*CrysAlis RED*; Oxford Diffraction, 2008)
 $T_{\min} = 0.013$, $T_{\max} = 0.156$
 6814 measured reflections

932 independent reflections
 919 reflections with $I > 3\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\max} = 26.5^\circ$, $\theta_{\min} = 3.4^\circ$
 $h = -6 \rightarrow 6$
 $k = -13 \rightarrow 13$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.012$
 $wR(F^2) = 0.030$
 $S = 1.25$
 932 reflections
 78 parameters
 1 restraint
 1 constraint
 Only H-atom coordinates refined

Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(I) + 0.0004I^2]$
 $(\Delta/\sigma)_{\max} = 0.013$
 $\Delta\rho_{\max} = 0.94$ e Å⁻³
 $\Delta\rho_{\min} = -0.53$ e Å⁻³
 Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
 Extinction coefficient: 48 (5)
 Absolute structure: Flack (1983), with 431 Friedel pairs
 Flack parameter: 0.01 (1)

Special details

Refinement. The refinement was carried out against all reflections. The conventional R -factor is always based on F . The goodness of fit as well as the weighted R -factor are based on F and F^2 for refinement carried out on F and F^2 , respectively. The threshold expression is used only for calculating R -factors *etc.* and it is not relevant to the choice of reflections for refinement.

The program used for refinement, JANA2000, uses the weighting scheme based on the experimental expectations, see `_refine_ls_weighting_details`, that does not force S to be one. Therefore the values of S are usually larger than the ones from the *SHELX* program.

supplementary materials

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pb1	0.5	0.47428 (2)	0.20948	0.01003 (7)
Pb2	0.5	0.84356 (2)	0.37011 (5)	0.01002 (8)
O1	0.5	0.4667 (5)	0.5354 (8)	0.0147 (15)
P1	0.5	0.34607 (15)	0.6573 (3)	0.0083 (5)
N1	0	-0.0035 (6)	0.4972 (8)	0.0103 (17)
O2	0	-0.0975 (5)	0.3893 (8)	0.0155 (14)
N2	0.5	0.7144 (6)	0.7547 (8)	0.0134 (18)
O3	0.2358 (7)	0.6561 (4)	0.2748 (5)	0.0109 (10)
O4	0.7011 (7)	0.7548 (3)	0.6898 (6)	0.0205 (11)
O5	-0.2007 (7)	0.0434 (3)	0.5539 (6)	0.0194 (11)
O6	0.5	0.6317 (6)	0.8801 (10)	0.033 (2)
H1	0.5	0.248 (4)	0.568 (10)	0.0099*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.00890 (14)	0.01098 (13)	0.01022 (12)	0	0	-0.00077 (12)
Pb2	0.00891 (15)	0.01137 (12)	0.00978 (12)	0	0	-0.00032 (9)
O1	0.012 (3)	0.020 (2)	0.013 (2)	0	0	0.002 (2)
P1	0.0048 (9)	0.0110 (8)	0.0091 (9)	0	0	0.0017 (5)
N1	0.011 (3)	0.013 (2)	0.007 (3)	0	0	0.000 (2)
O2	0.012 (3)	0.015 (2)	0.019 (2)	0	0	-0.006 (2)
N2	0.019 (4)	0.011 (3)	0.010 (3)	0	0	0.000 (2)
O3	0.0055 (18)	0.0151 (16)	0.0123 (17)	-0.0002 (15)	0.0003 (14)	-0.0033 (11)
O4	0.0123 (19)	0.0226 (16)	0.027 (2)	-0.0012 (15)	-0.0004 (16)	0.0012 (17)
O5	0.013 (2)	0.0235 (17)	0.0222 (16)	0.0054 (16)	0.0001 (18)	-0.0102 (17)
O6	0.049 (4)	0.030 (3)	0.019 (3)	0	0	0.015 (3)

Geometric parameters (\AA , $^\circ$)

Pb1—O1	2.346 (6)	Pb2—O4 ⁱⁱⁱ	2.707 (4)
Pb1—O1 ⁱ	3.042 (3)	Pb2—O5 ^{vii}	2.948 (4)
Pb1—O1 ⁱⁱ	3.042 (3)	Pb2—O5 ⁱ	2.782 (4)
Pb1—O3	2.418 (4)	Pb2—O5 ^{viii}	2.782 (4)
Pb1—O3 ⁱⁱⁱ	2.418 (4)	Pb2—O5 ^{ix}	2.948 (4)
Pb1—O4 ⁱⁱ	2.884 (4)	P1—O1	1.532 (6)
Pb1—O4 ^{iv}	2.884 (4)	P1—O3 ^x	1.530 (4)
Pb1—O6 ^v	2.881 (7)	P1—O3 ^{xi}	1.530 (4)
Pb1—O6 ⁱ	3.168 (4)	P1—H1	1.20 (5)
Pb1—O6 ⁱⁱ	3.168 (4)	N1—O2	1.249 (8)
Pb2—O2 ^{vi}	2.7756 (11)	N1—O5	1.258 (5)
Pb2—O2 ^{vii}	2.7756 (11)	N1—O5 ^{xii}	1.258 (5)

Pb2—O3	2.513 (4)	N2—O4	1.255 (5)
Pb2—O3 ⁱⁱⁱ	2.513 (4)	N2—O4 ⁱⁱⁱ	1.255 (5)
Pb2—O4	2.707 (4)	N2—O6	1.247 (9)
O1—Pb1—O1 ⁱ	114.73 (10)	O2 ^{vii} —Pb2—O5 ⁱ	64.37 (14)
O1—Pb1—O1 ⁱⁱ	114.73 (10)	O2 ^{vii} —Pb2—O5 ^{viii}	109.11 (14)
O1—Pb1—O3	80.31 (13)	O2 ^{vii} —Pb2—O5 ^{ix}	110.88 (13)
O1—Pb1—O3 ⁱⁱⁱ	80.31 (13)	O3—Pb2—O3 ⁱⁱⁱ	69.28 (12)
O1—Pb1—O4 ⁱⁱ	91.22 (13)	O3—Pb2—O4	101.25 (12)
O1—Pb1—O4 ^{iv}	91.22 (13)	O3—Pb2—O4 ⁱⁱⁱ	74.85 (12)
O1—Pb1—O6 ^v	147.26 (18)	O3—Pb2—O5 ^{vii}	169.07 (12)
O1—Pb1—O6 ⁱ	66.48 (13)	O3—Pb2—O5 ⁱ	109.08 (12)
O1—Pb1—O6 ⁱⁱ	66.48 (13)	O3—Pb2—O5 ^{viii}	83.30 (11)
O1 ⁱ —Pb1—O1 ⁱⁱ	125.41 (13)	O3—Pb2—O5 ^{ix}	110.97 (11)
O1 ⁱ —Pb1—O3	52.92 (13)	O3 ⁱⁱⁱ —Pb2—O4	74.85 (12)
O1 ⁱ —Pb1—O3 ⁱⁱⁱ	116.57 (13)	O3 ⁱⁱⁱ —Pb2—O4 ⁱⁱⁱ	101.25 (12)
O1 ⁱ —Pb1—O4 ⁱⁱ	130.14 (13)	O3 ⁱⁱⁱ —Pb2—O5 ^{vii}	110.97 (11)
O1 ⁱ —Pb1—O4 ^{iv}	69.42 (12)	O3 ⁱⁱⁱ —Pb2—O5 ⁱ	83.30 (11)
O1 ⁱ —Pb1—O6 ^v	63.03 (9)	O3 ⁱⁱⁱ —Pb2—O5 ^{viii}	109.08 (12)
O1 ⁱ —Pb1—O6 ⁱ	58.09 (14)	O3 ⁱⁱⁱ —Pb2—O5 ^{ix}	169.07 (12)
O1 ⁱ —Pb1—O6 ⁱⁱ	171.25 (15)	O4—Pb2—O4 ⁱⁱⁱ	47.35 (12)
O1 ⁱⁱ —Pb1—O3	116.57 (13)	O4—Pb2—O5 ^{vii}	68.85 (11)
O1 ⁱⁱ —Pb1—O3 ⁱⁱⁱ	52.92 (13)	O4—Pb2—O5 ⁱ	133.06 (12)
O1 ⁱⁱ —Pb1—O4 ⁱⁱ	69.42 (12)	O4—Pb2—O5 ^{viii}	174.94 (11)
O1 ⁱⁱ —Pb1—O4 ^{iv}	130.14 (13)	O4—Pb2—O5 ^{ix}	94.59 (12)
O1 ⁱⁱ —Pb1—O6 ^v	63.03 (9)	O4 ⁱⁱⁱ —Pb2—O5 ^{vii}	94.59 (12)
O1 ⁱⁱ —Pb1—O6 ⁱ	171.25 (15)	O4 ⁱⁱⁱ —Pb2—O5 ⁱ	174.94 (11)
O1 ⁱⁱ —Pb1—O6 ⁱⁱ	58.09 (14)	O4 ⁱⁱⁱ —Pb2—O5 ^{viii}	133.06 (12)
O3—Pb1—O3 ⁱⁱⁱ	72.44 (12)	O4 ⁱⁱⁱ —Pb2—O5 ^{ix}	68.85 (11)
O3—Pb1—O4 ⁱⁱ	171.11 (12)	O5 ^{vii} —Pb2—O5 ⁱ	81.63 (12)
O3—Pb1—O4 ^{iv}	109.00 (12)	O5 ^{vii} —Pb2—O5 ^{viii}	106.42 (11)
O3—Pb1—O6 ^v	73.43 (14)	O5 ^{vii} —Pb2—O5 ^{ix}	66.57 (11)
O3—Pb1—O6 ⁱ	72.12 (14)	O5 ⁱ —Pb2—O5 ^{viii}	45.92 (11)
O3—Pb1—O6 ⁱⁱ	134.56 (15)	O5 ⁱ —Pb2—O5 ^{ix}	106.42 (11)
O3 ⁱⁱⁱ —Pb1—O4 ⁱⁱ	109.00 (12)	O5 ^{viii} —Pb2—O5 ^{ix}	81.63 (12)
O3 ⁱⁱⁱ —Pb1—O4 ^{iv}	171.11 (12)	Pb1—O1—P1	126.8 (3)
O3 ⁱⁱⁱ —Pb1—O6 ^v	73.43 (14)	O1—P1—O3 ^x	109.20 (18)
O3 ⁱⁱⁱ —Pb1—O6 ⁱ	134.56 (15)	O1—P1—O3 ^{xi}	109.20 (18)
O3 ⁱⁱⁱ —Pb1—O6 ⁱⁱ	72.12 (14)	O1—P1—H1	113 (3)
O4 ⁱⁱ —Pb1—O4 ^{iv}	68.18 (11)	O3 ^x —P1—O3 ^{xi}	112.8 (2)
O4 ⁱⁱ —Pb1—O6 ^v	115.45 (13)	O3 ^x —P1—H1	106.4 (16)
O4 ⁱⁱ —Pb1—O6 ⁱ	102.09 (13)	O3 ^{xi} —P1—H1	106.4 (16)

supplementary materials

O4 ⁱⁱ —Pb1—O6 ⁱⁱ	41.64 (14)	O2—N1—O5	120.4 (3)
O4 ^{iv} —Pb1—O6 ^v	115.45 (13)	O2—N1—O5 ^{xii}	120.4 (3)
O4 ^{iv} —Pb1—O6 ⁱ	41.64 (14)	O5—N1—O5 ^{xii}	119.3 (5)
O4 ^{iv} —Pb1—O6 ⁱⁱ	102.09 (13)	Pb2 ^{xiii} —O2—Pb2 ^{xiv}	153.8 (2)
O6 ^v —Pb1—O6 ⁱ	121.12 (11)	Pb2 ^{xiii} —O2—N1	101.78 (11)
O6 ^v —Pb1—O6 ⁱⁱ	121.12 (11)	O4—N2—O4 ⁱⁱⁱ	120.0 (5)
O6 ⁱ —Pb1—O6 ⁱⁱ	117.19 (15)	O4—N2—O6	120.0 (3)
O2 ^{vi} —Pb2—O2 ^{vii}	153.82 (14)	O4 ⁱⁱⁱ —N2—O6	120.0 (3)
O2 ^{vi} —Pb2—O3	68.37 (13)	Pb1—O3—Pb2	108.96 (14)
O2 ^{vi} —Pb2—O3 ⁱⁱⁱ	137.63 (13)	Pb1—O3—P1 ⁱ	111.9 (2)
O2 ^{vi} —Pb2—O4	115.10 (14)	Pb2—O3—P1 ⁱ	129.5 (2)
O2 ^{vi} —Pb2—O4 ⁱⁱⁱ	69.02 (14)	Pb1 ^{xv} —O4—Pb2	123.29 (14)
O2 ^{vi} —Pb2—O5 ^{vii}	110.88 (13)	Pb1 ^{xv} —O4—N2	101.0 (3)
O2 ^{vi} —Pb2—O5 ⁱ	109.11 (14)	Pb2—O4—N2	94.7 (3)
O2 ^{vi} —Pb2—O5 ^{viii}	64.37 (14)	Pb2 ^{xiii} —O5—Pb2 ^x	151.56 (16)
O2 ^{vi} —Pb2—O5 ^{ix}	44.54 (13)	Pb2 ^{xiii} —O5—N1	93.1 (3)
O2 ^{vii} —Pb2—O3	137.63 (13)	Pb2 ^x —O5—N1	95.3 (3)
O2 ^{vii} —Pb2—O3 ⁱⁱⁱ	68.37 (13)	Pb1 ^{xvi} —O6—N2	171.0 (5)
O2 ^{vii} —Pb2—O4	69.02 (14)	Pb1 ^{xvi} —O6—O4	148.84 (17)
O2 ^{vii} —Pb2—O4 ⁱⁱⁱ	115.10 (14)	Pb1 ^{xvi} —O6—O4 ⁱⁱⁱ	148.84 (17)
O2 ^{vii} —Pb2—O5 ^{vii}	44.54 (13)		

Symmetry codes: (i) $-x+1/2, -y+1, z-1/2$; (ii) $-x+3/2, -y+1, z-1/2$; (iii) $-x+1, y, z$; (iv) $x-1/2, -y+1, z-1/2$; (v) $x, y, z-1$; (vi) $x, y+1, z$; (vii) $x+1, y+1, z$; (viii) $x+1/2, -y+1, z-1/2$; (ix) $-x, y+1, z$; (x) $-x+1/2, -y+1, z+1/2$; (xi) $x+1/2, -y+1, z+1/2$; (xii) $-x, y, z$; (xiii) $x-1, y-1, z$; (xiv) $x, y-1, z$; (xv) $-x+3/2, -y+1, z+1/2$; (xvi) $x, y, z+1$.

Fig. 2

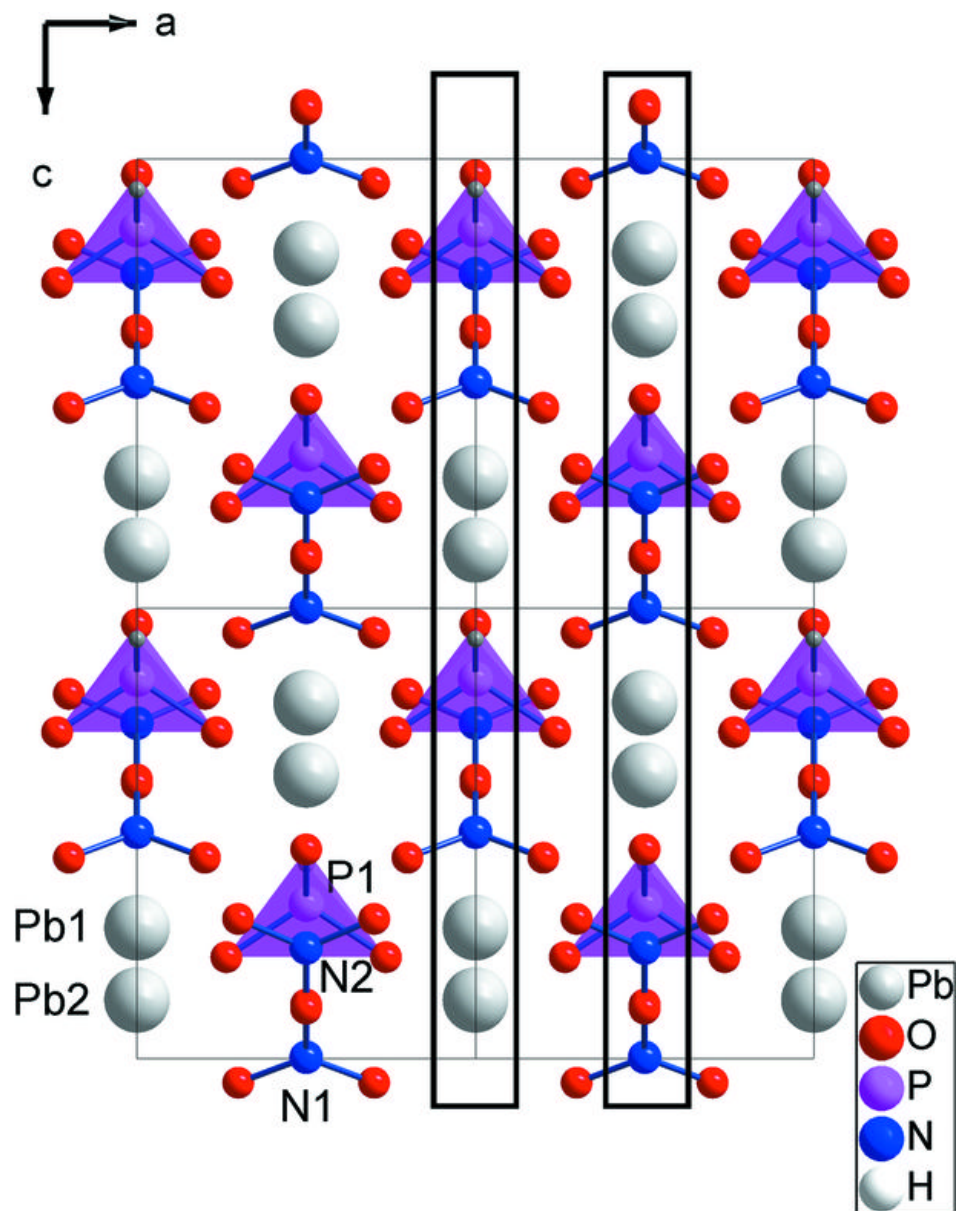


Fig. 3

